

DC-Arc Optical Emission Spectrography

Direct-current-arc optical emission spectrography (DC-arc) is primarily an element survey technique used to determine elemental composition at the trace, minor, and major levels, usually in solid samples (occasionally in liquid samples). The method can be applied to the analysis of more than 65 elements. Because visual comparisons of spectra are made, DC-arc is essentially a qualitative or semiquantitative method with accuracy of about 50% relative. It provides comprehensive information on trace elements or overall sample composition in a relatively short time.

Principle of Technique

The material to be analyzed is placed in a graphite cup electrode and burned in a dc arc. The elements constituting the material are vaporized into the arc plasma, and the emitted light is directed by a lens system into an optical spectrograph. There it is dispersed into a spectrum and photographed. Each element emits its own characteristic optical spectrum that varies in complexity depending on atomic structure. For example, sodium emits only a few lines while uranium emits a very complex spectrum. The overall complexity of the spectrum of a sample, therefore, is a function of the elements present and their concentration. In making a visual determination, the optimum characteristic lines are chosen and the concentration is determined by comparison of the line intensity with its counterpart in a series of matrix-matched standards.

Samples

Form. The method is typically applied to solids and sometimes liquids.

Size. Although milligram or submilligram samples can be analyzed, it is recommended that gram quantities be submitted, if possible. This will ensure preparation of a representative sample for analysis.

Preparation. Solid samples as powder, fine turnings, or chips are mixed with graphite and transferred to a graphite crater electrode. Liquid samples are evaporated to dryness. In cases where only minor or trace elements are of interest, $\text{Ga}(\text{NO}_3)_3$ is added to collect these elements as the solution evaporates. The residual salt is converted to Ga_2O_3 , mixed with graphite, and transferred to the electrode. Organic samples are usually decomposed (wet-ashed) followed by the collection of trace elements in Ga_2O_3 .

Examples of Applications

- Determination of trace elements in metals, chemical compounds, plastics, and aqueous or organic solutions.
- Identification of the elemental composition of alloys and unknown materials such as corrosion deposits.
- Determination of the approximate composition of geological materials such as rocks and minerals.
- Semiquantitative determination of metallic and metalloid impurities in metallic alloys.



A portion of the emission lines from a stainless-steel standard in DC-arc determinations.

Limitations

Detection limits are usually in the parts-per-million range. They vary depending on the complexity of the matrix spectrum, and the preconcentration procedures applied (e.g., solution evaporation or sample decomposition). Elements that cannot be determined include the halogens, noble gases, hydrogen, carbon, nitrogen, oxygen, sulfur, selenium, and tellurium. DC-arc cannot provide structural information or determine oxidation states of atomic constituents. DC-arc is best used for qualitative or semiquantitative measurements. Other methods are generally superior for quantitative measurements.

Estimated Analysis Time

Usually 6 to 8 h per sample is needed for semiquantitative determinations. Samples that require extensive preparation or whose spectral comparisons involve complex spectra can take more time, typically 10 to 16 h.

Related Techniques

Inductively-coupled plasma atomic emission spectroscopy (ICP-AES), atomic absorption spectroscopy, and DC-plasma optical emission spectroscopy are available for the quantitative determination of elemental composition of solutions or dissolved solids. Detection limits are in the parts-per-million range.

Inductively-coupled plasma mass spectrometry (ICP-MS) applications are similar to those for ICP-AES with detection limits ranging from parts per trillion to parts per billion. ICP-MS can also provide isotopic information if required.

X-ray fluorescence spectrometry is available for the nondestructive, semiquantitative elemental analysis of solids. Detection levels are typically in the parts-per-million range, except for elements lighter than silicon.